STEROLS AND BILE ACIDS IN URBAN AND RURAL SOILS AS FAECAL MARKERS OF LAND-USE SINCE THE BRONZE AGE

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Section A18
Poster 508

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Introduction and aims

Faecal biomarkers (C27, C28 and C29 coprostanols and bile acids) found in soils and sediments have been proposed as direct indicators of human presence or of breeding since these compounds are formed in the intestinal tracts of higher mammals (Elhmmali et al, 2000, Bull et al., 2002).

The goal of this study is to test the potentialities of fecal biomarkers in soils and sediments to resolve, at the field-scale, the strategies of land use. For this purpose, we designed our study in three steps:

i) characterization of faecal biomarkers imprints of source animals,

ii) evaluation of the faecal biomarkers stability through the analysis of samples of different age,

iii) examination of the distribution of faecal biomarkers in a wide diversity of contexts under different type of soils, different land-uses and degrees of anthropization.

Methodology

Dried and crushed samples (~5 g) were extracted using accelerated solvent extraction with CH_2Cl_2:MeOH (1:1 v/v; ASE 200 Dionex®). Total extract was fractionated into neutral and acidic compounds using solid phase extraction on Aminopropyl Bond Elute© phase according to Jacob et al. (2005). Acid fractions were methylated (anhydrous MeOH and acetyl chloride). Then, sterols and acidic fractions were further derivatized by BSTFA. Standards (5α-cholestane and nor-deoxycholic acid) were added prior to GC-MS analysis.

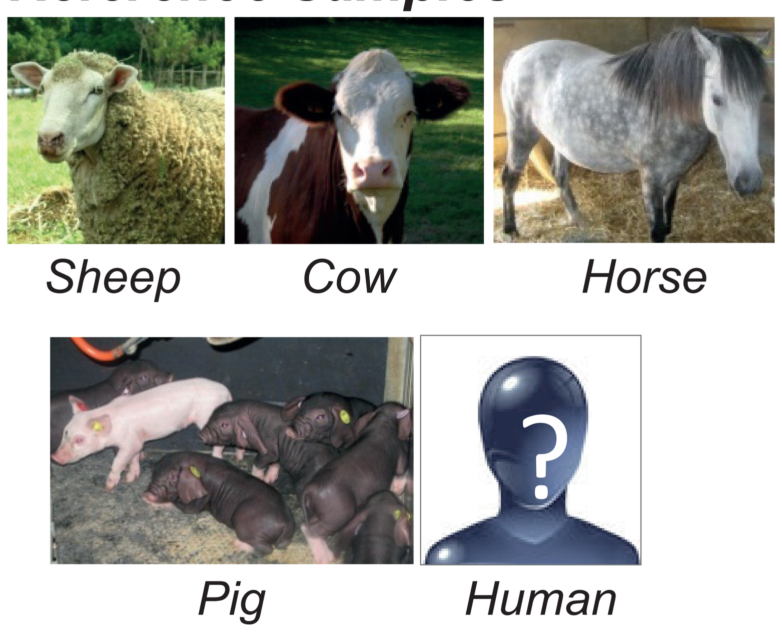
GC-MS details for sterols

	compound	MM	major m/z
Cp	coprostanol	388	215, 257, 355,370
epi-Cp	epi-coprostanol	388	215, 257, 355,370
Chl	cholesterol	386	368, 353, 329, 255
Cln	cholestanol	388	215, 355, 455, 460
mCp	methyl-coprostanol	402	215, 257, 369, 384
eCp	ethyl-coprostanol	416	215, 257, 383, 398
epi-eCp	epi-ethyl-coprostanol	416	215, 257, 383, 398
Sterl	stigmasterol	412	255, 394, 379
Stanl	stigmastanol	416	215, 257, 383, 398
Sit	sitosterol	414	396, 357, 381, 255

GC-MS details for bile acids

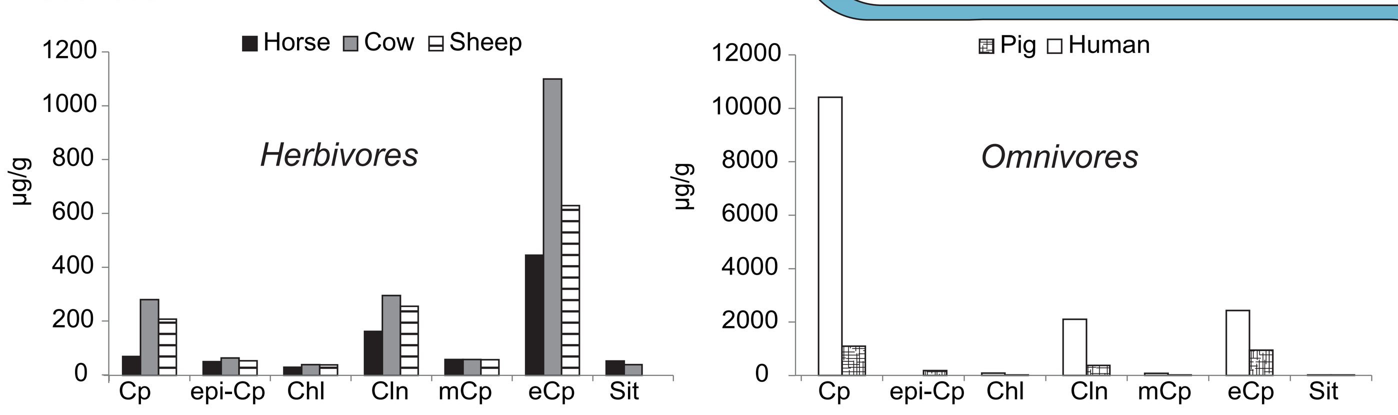
	compound	MM	major m/z
LC	lithocholic acid	462	215,257,357,372
DOC	deoxycholic acid	550	208,255,345,370
C	cholic acid	638	253,343,368,458
CDOC	chenodeoxycholic acid	550	255,355,370,460
HDOC	hyodeoxycholic acid	550	255,355,370,460
UDOC	ursodeoxycholic acid	550	255,355,370,460

Reference Samples



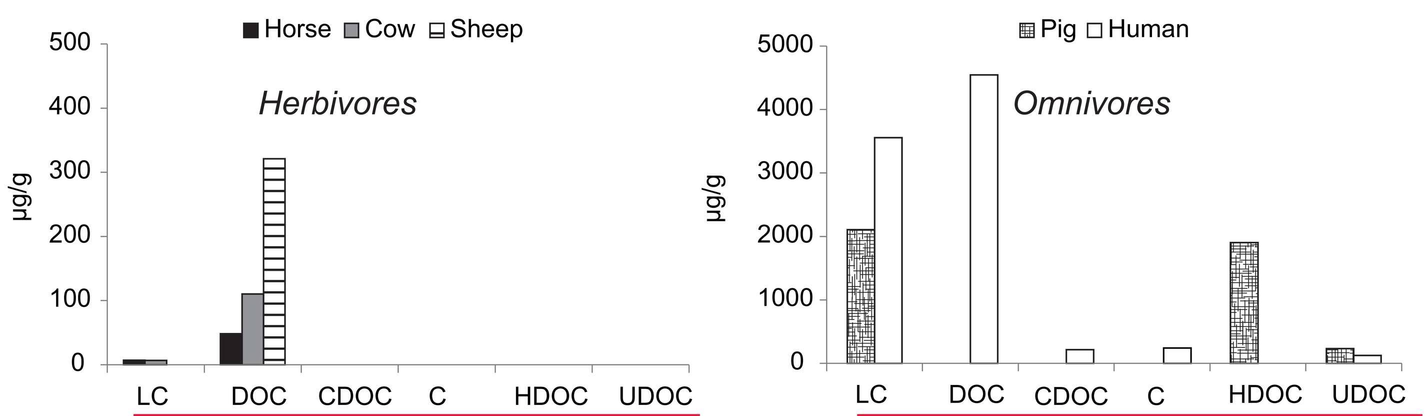
Animal imprints

Sterols



Significant differences in sterol imprints between herbivores and omnivores

Bile Acids



Sheeps show different bile acids imprint than cows and horses

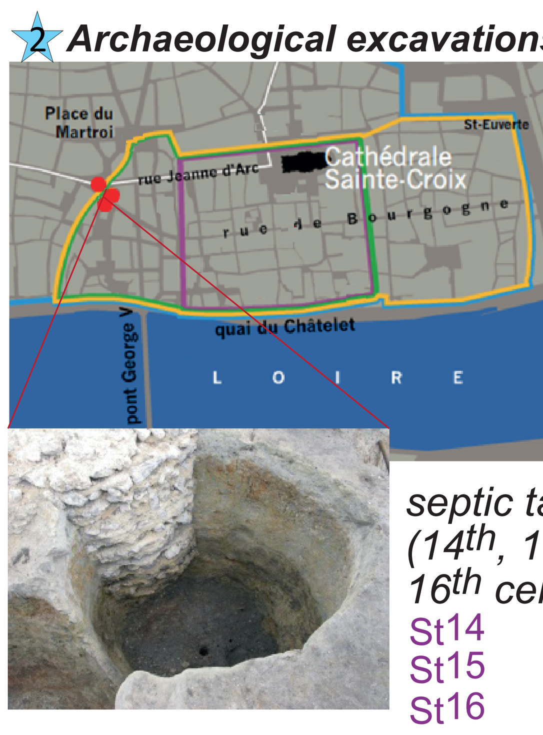
Pig and Human can be distinguished by bile acids imprints

Collection Samples

1 Septic tank

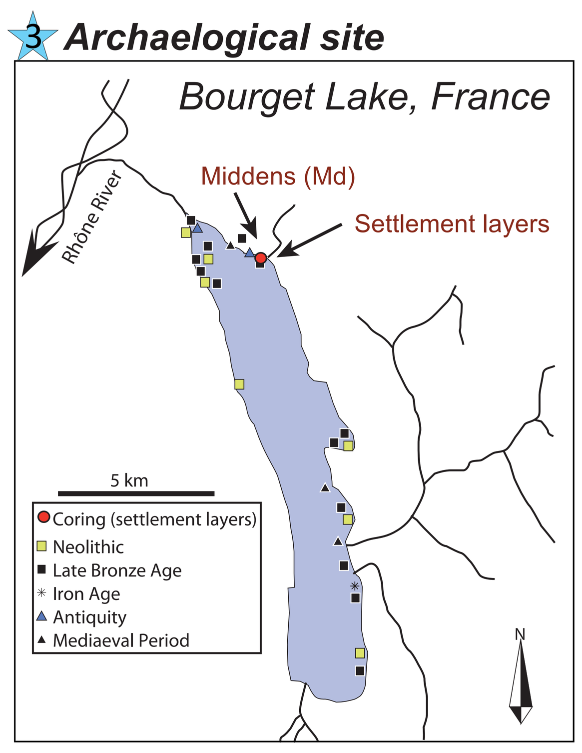
19th century St19

2 Archaeological excavations

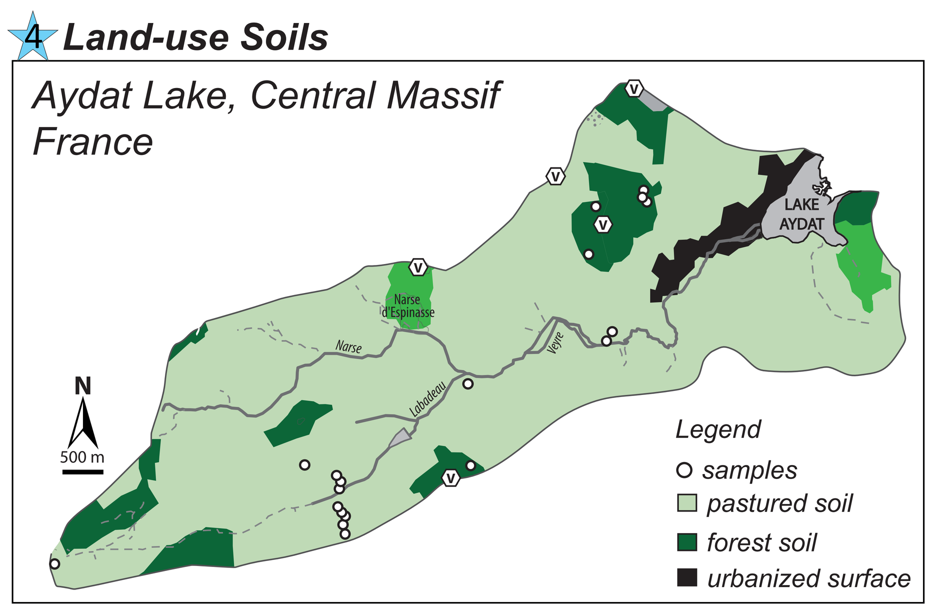


septic tanks (14th, 15th and 16th century)
St14
St15
St16

3 Archaeological site



4 Land-use Soils

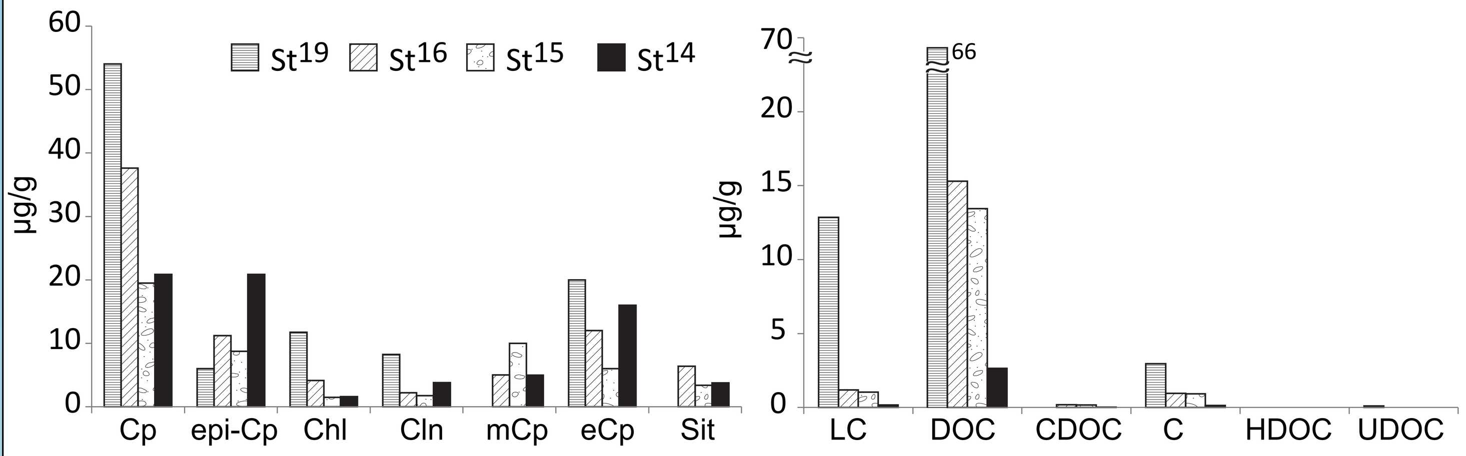


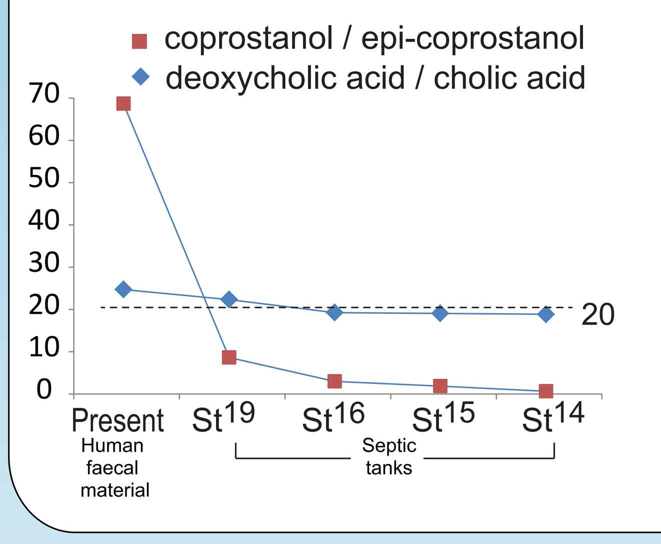
3 middens Md
25 settlement layers
Bronze Age

10 soils under pasture Ps
6 soils under forests Fs

Faecal biomarkers preservation in septic tanks

Once demonstrated that animals have different imprints, we are interested in evaluating the preservation of faecal biomarkers in sedimentary profiles. Their distribution in septic tanks active during the 19th, 16th,15th and 14th centuries are compared (St19, St16, St15 and St14, respectively).





- In septic tanks, sterols and bile acids decrease with time. For bile acids, diagenesis undistinctively affects compounds whereas sterols are inequally affected.
- This is futher evidenced by the deoxycholic/cholic acid ratio that remains constant (~20) whereas the coprostanol/epi-coprostanol ratio decreases with time.

Variability of faecal biomarker imprints

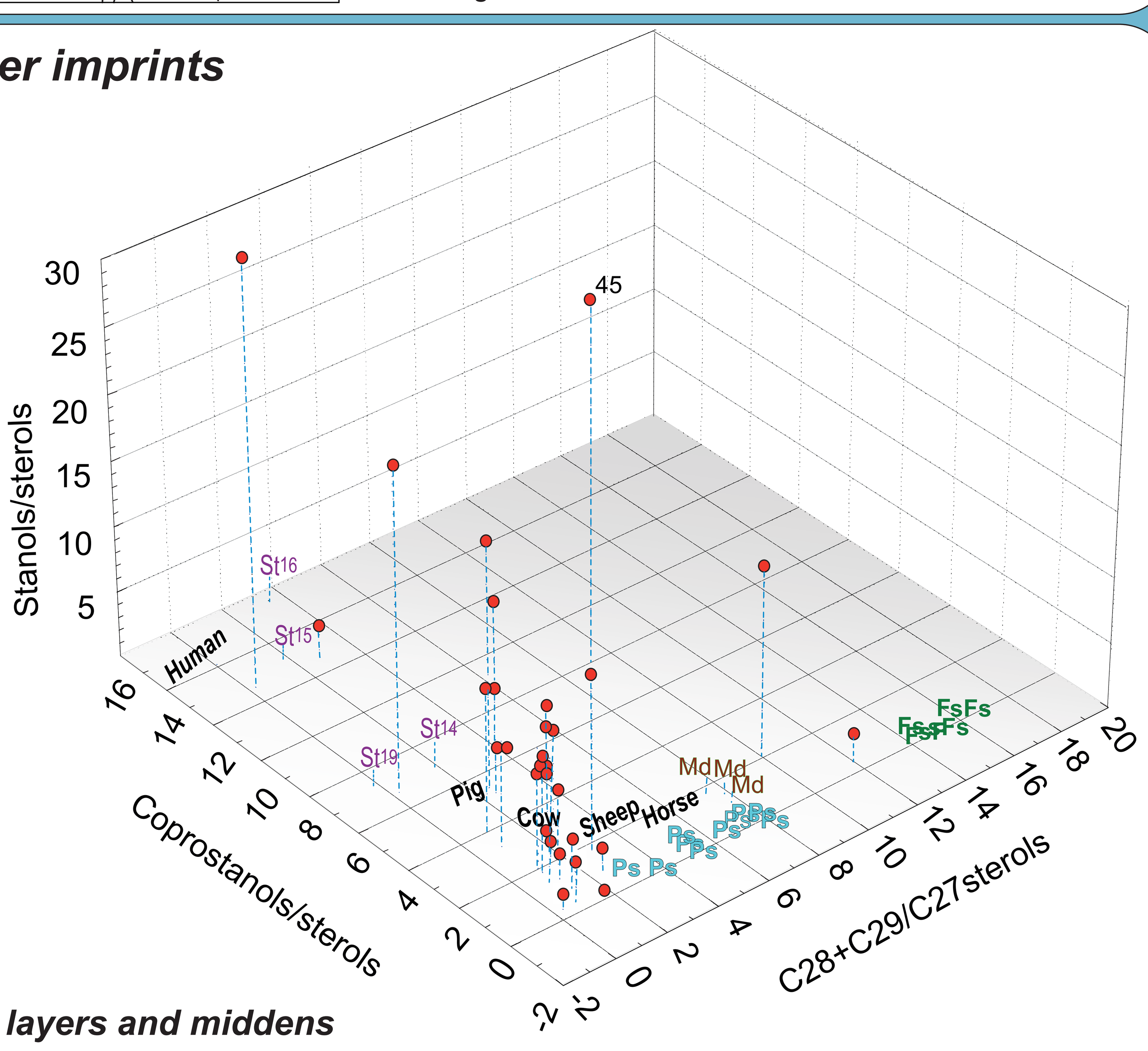
Sterols index

Sterol ratios were used to identify the likely source and degradation state of faecal biomarkers in samples.

Recent samples such as the fresh faecal material (*Reference samples*), pastured and florest soils (*Aydat catchment*) are spread along coprostanol/sterol and C28+C29/C27 sterols axis.

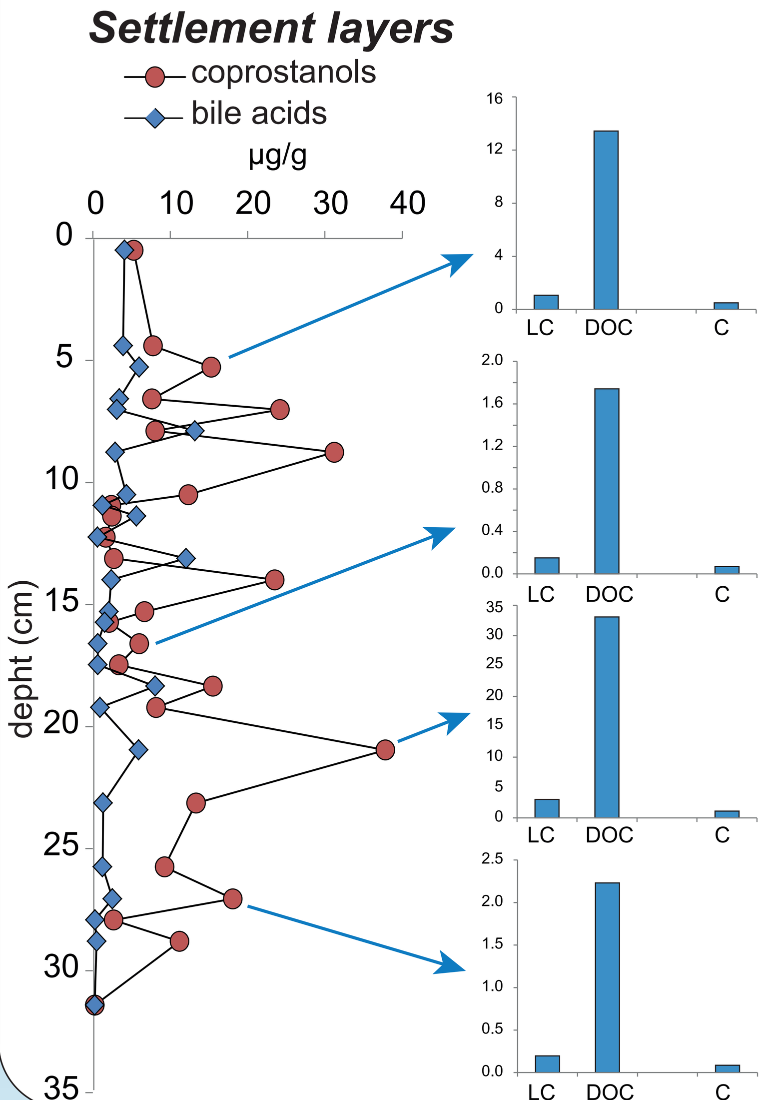
Archeological samples from septic tanks and middens have low stanols/sterols index whereas settlement layers have high values, indicating the sterols transformation into stanols (reduction) through time.

Diagenesis could entail coprostanols reduction thus masking the source imprints (Mermoud et al., 1984; Rushdi et al., 2006).

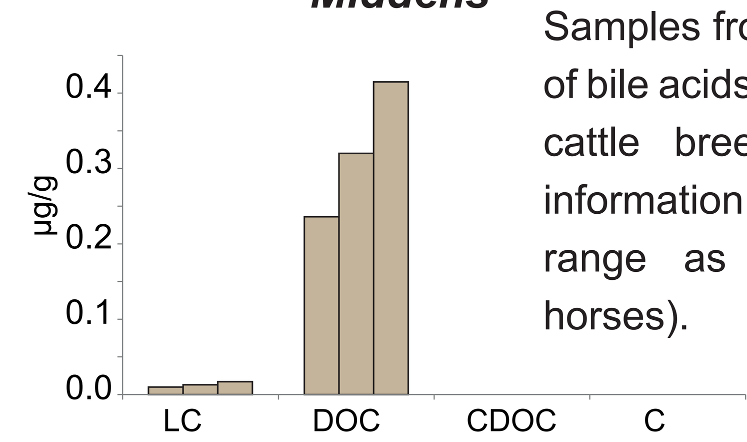


Bile acids in Bronze Age settlement layers and middens

Settlement layers



Middens



Samples from middens contain low amounts of bile acids (only LCA and DCA) indicative of cattle breeding. These data corroborate information obtained sterol data (in the same range as pastures, cows, sheeps and horses).

Coprostanols and bile acids vary across the profile in settlement layers. However, all settlement layers exhibit similar bile acids distribution with a deoxycholic/cholic acid ratio are greater than 20. This is indicative of a major contribution from Human feces but is contradictory to information retrieved from sterols that point to a significant contribution of cattle (cf. 3d graph). Nevertheless, sterols are more prone to degradation that can bias the original imprint.

Conclusions

- Significant differences in faecal biomarker imprints between allow distinguishing between animal sources: sheeps, cows, horses, pigs and Humans.
- Discrimination based on presence/absence of bile acids appear more effective than that based on sterols relative abundances.
- Bile acids proportion remain constant with time, and more stable than sterols.
- Combination of sterols and bile acids over a wide range contexts demonstrate their complementarity and efficiency in providing information on former land-uses.

Acknowledgments:

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- Rushdi et al., 2006. Environmental Geology 50, 1171 - 1181

Bile acids in soils under pastures and forests

Soils under pastures contain notable amounts of lithocholic and deoxycholic acids that testifies to their present/recent use for cattle breeding or to manuring practises. Bile acids were absents in soils under forest.